Fabrication and Properties of Er-Doped Nanocrystalline Phase-Seperated Optical Fibers

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Abstract. *The fabrication of Er-doped ZrO_2-based nanocrystalline phase-separated optical fiber preforms using the MCVD and solution doping techniques is presented and discussed in details in the paper. Preforms with nearly transparent cores containing Er-doped nanocrystals with dimensions mainly within 20 – 80 nm were prepared. Fibers drawn from the preforms exhibit good luminescent properties around 1550 nm suitable for applications in fiber lasers and amplifiers.*

Keywords

Er-doping, nano-crystalline optical fiber, phase-separated glass.

1. Introduction

Recently, substantial research effort has been focused on the preparation of rare-earth (RE) doped fibers and their implementation for the development of fiber lasers and amplifiers. This research has primarily dealt with Er-doped fibers (EDFs) based on different glass materials, such as doped silica, phosphates, tellurides etc. It has been aimed at tailoring RE doping levels, emission properties of RE, fiber losses etc. Different glass materials used as matrices for the immobilisation of REs have shown their own advantages and limitations when considered for device applications [1]. However, taking into account ruggedness and compatibility of silica-based fibers with present telecommunication systems, one can understand reasons why researchers have primarily focused to materials and preparation routes for silica-based fibers. A number of approaches and materials have been investigated for tuning spectral properties of RE-doped silica fibers such as broadening of the emission bandwidth of EDFs which can be employed for amplifying more channels in wavelength division multiplexing (WDM) systems.

It is evident that nowadays such investigations can hardly bypass employing of nanomaterials. It is well known that nanoparticles can induce wide spectrum of size-dependent properties. This was one of the reason why researchers have investigated the incorporation of different types of Er-oxide nanocrystallites into different glass hosts with the aim to tailor their spectroscopic and lasing characteristics [2], [3], [4], [5]. The preparation of Er-doped nanocrystalline phase-separated materials represents important modification of the approaches mentioned above because it provides us not only with single nanoparticles but with nanostructured blocks. Besides the targeted broader emission around 1,5 μm which coincides with the lowest attenuation window of silica glasses, such materials could also exhibit improved luminescence properties and find applications in 3D displays, upconversion lasers etc. [6].

Among various techniques available for the preparation of RE-doped nanocrystalline phase-separated multicomponent core materials such as co-sputtering [7], laser ablation [8], ion-implantation [9], pyrolysis [10], sol-gel technique [11] and recently developed direct nanoparticle deposition (DND) technique [12], particularly the double-crucible technique has been employed for this purpose [13]. However, in our research we have been dealing with the preparation of RE-doped nanocrystalline phase-separated materials from high-silica based glasses which can't be obtained by the double crucible technique. Thus we have decided to prepare them by using the Modified Chemical Vapor Deposition (MCVD) combined with the solution doping technique [14].

In order to prepare such materials it is indispensable to incorporate some oxides into glass controlling nucleation processes in it, like CaO, Y_2O_3, ZrO_2 etc. Recently, several papers have been published [15], [16],

[17] dealing with the nucleation control in silica-based fiber preforms by using CaO, ZrO_2,Y_2O_3 or Yb_2O_3 doped glass matrices incorporating Er ions and prepared by the MCVD and solution doping techniques.

In our investigation we have employed ZrO_2 combined with BaO for fabricating preforms of Er-doped nanocrystalline phase-separated silica-based optical fibers suitable for fiber lasers and amplifiers. Our motivation for using ZrO_2 for such fabrications is related to facts that this oxide supports nucleation in glass, lowers the glass phonon energy and enhances the probability of radiative transition [18]. This will essentially leads to display wider emission and absorption bandwidths and allowing more channels to be amplified as compared to materials with a lower refractive index. This can be explained with the Fuchtbauer-Ladenberg relationship [19] and Judd-Ofelt theory [20].

Doping of Li and Al is necessary to achieve Li_2O-Al_2O_3-SiO_2 glass composition which is extremely interesting because of the high thermal-mechanical strength, near zero thermal expansion and transparency. Addition of small amounts of zirconium oxide was chosen as host matrix for erbium ions. Incorporation of ZrO_2 in glass matrix lowers the phonon energy (470 cm -1) which enhances the number and the probability of radiative transitions in Er-doped zirconia host nanocrystals. Simultaneously, Zr exhibit good optical transparency, chemical stability, high refractive index ($n = 2,15$ @ 633 nm) and has a higher boiling temperature than the drawing temperature of silica fiber. Zirconia nanocrystals formation is expected to shift emission wavelengths of Er ions by upconversion mechanisms and enhances the luminescence efficiency depending on their nanometer dimension. Incorporation of BaO will serve as network modifier, plays an important role as a catalyst during nucleation process besides reducing the melting temperature of the glass mixture.

In this paper detailed preparation of the preforms by the MCVD and solution-doping techniques is described and the formation of nanocrystalline particle within the phase-separatedpreform core is presented. Optical properties of fibers drawn from the preforms are also discussed.

2. Preparation of Preforms

The fabrication of Er-doped nanocrystalline phase-separated optical fibers employed three main processing stages, namely the preform fabrication, heat treatment of the fabricated preform followed by the fiber drawing. An extensive set of experiments have been made in order to determine key processing parameters necessary for preparing preforms with tailored properties. These parameters include a temperature range of the deposition of the porous core layer, concentrations of dopants in the soaking solution, temperatures at the dehydration and consolidation steps and the temperature of the thermal processing of the preform.

The preform fabrication was carried out on a standard MCVD setup (Special Gas Controls, GB). The process started with the MCVD deposition of cladding layers inside a rotating substrate silica tube (F-300, an outer diameter of 18 mm, wall thickness of 1,4 mm). The deposition temperature was monitored by an optical pyrometer moving synchronously with an oxy-hydrogen burner used for heating the tube.

Following the cladding layers, a porous core layer containing either pure silica or silica doped with a small amount of P_2O_5 ($\sim$1 mol%) was deposited. One can expect that the temperature during the deposition of the porous core layer is important for controlling the layer porosity and its adhesion to the tube wall. It is evident that a low temperature gives us a way how to prepare a layer with a high porosity and consequently with high dopant concentrations but also it could increase the probability of the layer delamination (peeling off) from the tube wall due to contact with the soaking solution. In a set of experiments we have determined that temperatures in a range of $1250 - 1300$ °C are suitable for preparing porous layers with a sufficient porosity and adhesion. In all experiments the deposition temperature of the porous layer was maintained with a precision of 5 °C along the tube length in order to obtain an uniform porous layer.

The tube with the deposited porous layer was then dipped into an aqueous solution for about 40 minutes. The solution contained water-soluble salts namely $LiNO_3$ (99,995 % Merck, Germany), $AlCl_3$ (99,99 % from MaTeck GmbH), $BaCl_2{\cdot}2H_2O$ (99,999 %, Aldrich), $ZrOCl_2{\cdot}xH_2O$ (99,99 %, Aldrich) and $ErCl_3{\cdot}6H_2O$ (99,995 %, Aldrich). A final composition of the soaking solution used for the preparation of phase-separated preforms was determined on the basis of a set of trial experiments in which the composition of the soaking solutions was varied and compositions of the corresponding preform samples were measured by using Electron Microprobe Analysis (EMA). The composition of the soaking solution used for the preparation of preforms for this paper was selected as 1,25 M $AlCl_3$, 0,035 M $LiNO_3$, 0,5 M $BaCl_2$, 0,035 M $ZrOCl_2$ and 0,03 M $ErCl_3$.

The soaked layer was dried in a flow of air for one hour followed by dehydration for 30 minutes in the presence of chlorine at temperatures around $650 - 700$ °C. This drying temperature was optimized in order to prevent the evaporation of $LiNO_3$ soaked into the porous layer. Chlorine drying was performed to

reduce the OH level, presence of which increases the attenuation of fiber around 1,3 μm wavelength region.

Subsequently, the porous layer was consolidated to clear glass in the presence of oxygen gradually by increasing the temperature from 1000 to 1800 °C with a step of 100 °C. The gradual increase of the temperature during the consolidation of the porous layer in presence of oxygen was found to be important to prevent any sort of Al_2O_3 crystallization on the core-cladding boundary which can enhance scattering losses and degrade light guiding properties. In the consolidation step a proper concentration of BaO in glass was also important since besides acting as good nucleating agent, it prevents cracking caused during the preform annealling stage as a result of volume expansion when ZrO_2 structural transformations occurs [21].

Finally, the tube with the prepared layers was collapsed at a temperature higher than 2000 °C to obtain a rod, the preform, of a diameter around 10,5 mm. After the collapsing step the fabricated preform was heat treated in an electrical furnace at around 1000 °C for about 5−6 hours. The heat-treatment of the preform is primarily responsible for the crystallization of nanoparticles and phase-separation processes in the preform core.

Fibers of diameter $125 \pm 0,5$ μm were drawn at a drawing tower provided with a graphite furnace Centorr, USA. The fibers were coated by a protective UV-curable acrylate jacket (De Solite) during drawing.

3. Characterisation of Preforms and Fibers

Refractive index profiles (RIPs) of the fabricated preforms were measured using a PK2600 preform analyzer (Photon Kinetics, USA). RIPs of the fibers were determined on an S14 fiber analyzer (York Technology, GB).

Scanning Electron Microscopy (SEM) was used for the identification of nanocrystalline particles on a thin slice of the polished preform sample f (thickness 1,5 mm). Transmission Electron Microscopy (Tecnai^{TM}G^2 F30) was employed for observing the phase separation in the preform core. For this purpose the cladding part of a short segment (of about 1 mm) of the preform was chemically etched out by a solution of hydrofluoric acid and the extracted core was crushed to powder that was analyzed. A suspension of the powder was applied onto a grid of the TEM device. The high sensitivity of TEM instrument (point to point resolution of 0,20 nm and line resolution of 0,14 nm) was suitable to detect the nanocrystalline particles generated (from 10 to 40 nm) within the preform core. Electron diffraction patterns were also measured on the powdered preform cores. To identify the presence of nanocrystalline particles within the fiber core, using HRTEM, it was necessary to extract the core (∼12 μm) from the 125 μm fiber. We found this selective extraction was difficult, so we adopted a special technique. Piece of short fiber sample was etched out precisely to expose the fiber core doped with Er. Then atleast 3 to 4 such short fiber samples were placed side by side and HRTEM was performed across the length of this bundle of sample to make sure we focus exactly in the preform core. To evaluate the composition of phase-separated particles the electron beam was focused on the particles and then focused in an area outside of the particles, when the energy dispersive X-ray analyses (EDX) data were taken. The nature of the particles was evaluated from their electron diffraction pattern.

Spectral attenuation losses of the prepared fibers in a range from 800 to 1600 nm were measured using the conventional cut-back technique on a set up with a white light source and a grating monochromator. The luminescence of Er in the prepared fibers was measured on a short piece of the fiber (a length ∼14 mm) that was spliced with a short piece of a standard single mode fiber (SMF) using the contra-propagative configuration. Such a short fiber length was selected to eliminate distortion by reabsorption and amplified spontaneous emission (ASE).

4. Result & Discussion

An example of the RIP of the fabricated preforms is shown in Fig. 1, the corresponding RIP of the fiber in Fig. 2. These figures show that the both profiles do not substantially differ. Measurements of RIPs of the preforms have shown that the numerical apertures of the preform cores are within $0,16 \pm 0,02$. No noticeable changes in RIPs of the preforms were observed even after the preform annealing stage. From Fig. 2 it is evident that this fiber has a core diameter of about 12 μm with respect to 125 μm cladding diameter. Thus, the fiber is multimode around wavelength of 1550 nm.

Results of the TEM analysis of a sample of the preform core are presented in Fig. 3. One can recognize two distinct regions on the photo; one rich in Al_2O_3 (part with the grey color) and another rich in ZrO_2.

The electron diffraction pattern of the powder in Fig. 4 indicates crystalline nature of the particles. Results of the SEM shown on the inset in Fig. 4 give us the direct evidence of the presence of nanocrystalline particles in the preform core with average dimensions ranging from $20-80$ nm. We have additionally carried out HRTEM analysis of fiber sample employing special technique and result indicates existence of nanocrys-

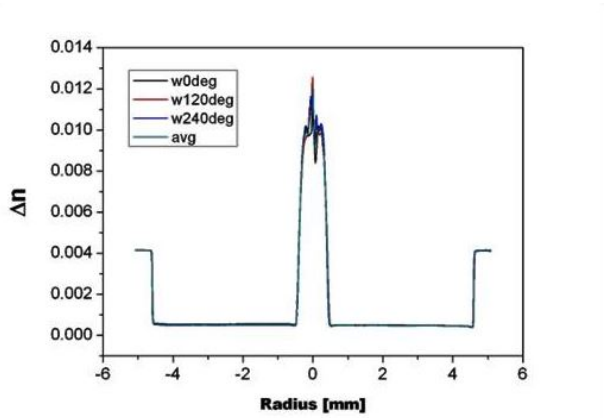

Fig. 1: Refractive-index profiles of the fabricated preform.

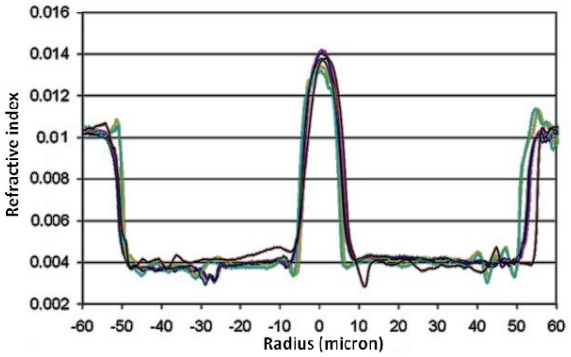

Fig. 2: Refractive-index profiles of the fabricated fiber.

talline particles (black dot inset in Fig. 5.), surviving the drawing temperature and tension. Average particle size determined during HRTEM analysis indicate particle size within $10-40$ nm which is within the range of our preform analysis result. The corresponding EDX data in Fig. 5 provide us with the local composition of the nanocrystalline particle region that contain signal of Zr, Er. Ba and Al.

Concentration profiles of oxide dopants within the preform core were determined by the EMA and presented in Fig. 6. These profiles reveal that maximally about 14 mol% of Al_2O_3 and 9600 ppm of Er^{3+} can be incorporated by using the developed approach. The corresponding concentrations of Zr and Ba are 0,9 and 0,2 mol%, respectively. The concentration of Li could not be measured since elements having atomic weight below 5 cannot be detected by the EMA.

Despite of the high contents of aluminum oxide in the preform core, the cores is nearly transparent. Additionally, the core-clad interface when investigated by optical microscope revealed no "star-like" pattern along the core-clad boundary related to high Al_2O_3 concentration, frequently observed in case of highly doped Al_2O_3 preforms [22].

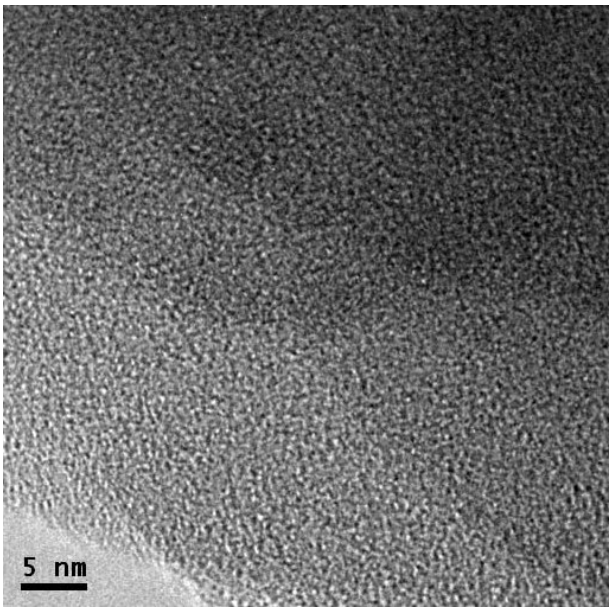

Fig. 3: TEM image taken on a sample of the preform core and showing phase separation.

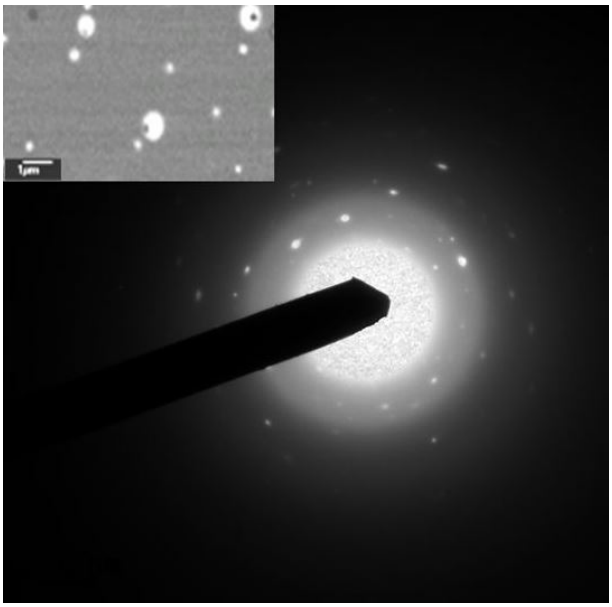

Fig. 4: Electron diffraction pattern indicating crystalline nature of sample; inset SEM image of nanoparticle reach preform core.

An example of the spectral attenuation curve of the prepared fiber is shown in Fig. 7. Two absorption bands around 980 and 1550 nm in Fig. 7 can be attributed to Er absorption. A concentration of Er in the fiber core was estimated from a height of the band around 980 nm. This concentration well agrees with the maximum Er concentration determined from Fig. 6. From the curve in Fig. 7 one can also conclude that the base-line losses of the fiber are high. They could be related to scattering losses of nanocrystalline particles in the fiber core. The observed nanocrystalline particles

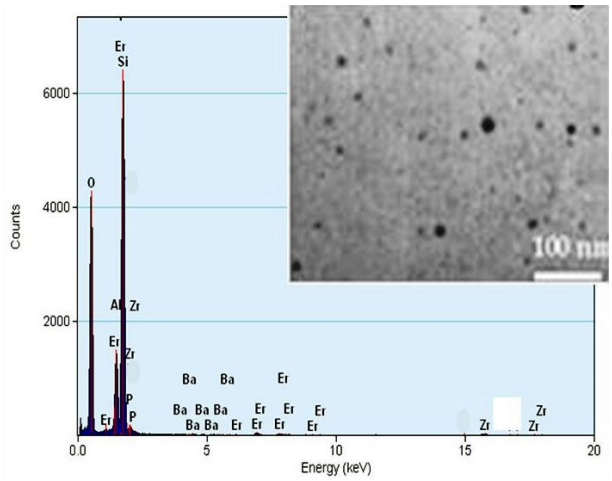

Fig. 5: HRTEM picture of the fiber core region containing nanocrystalline particle with EDX result.

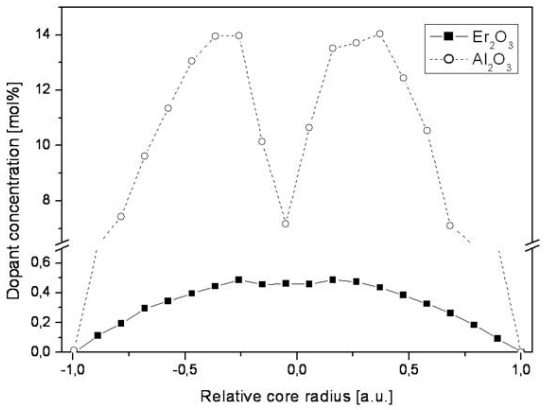

Fig. 6: Distributions of Al_2O_3 and Er_2O_3 through the preform core determined by EMA.

(size greater than 20 nm) in the fiber core is the reason of high base-line losses and indicate the presence of some inhomogeneities in the preform core, especially if the core-cladding boundary is not distorted [15].

When used pumping at 978 nm with a pump power of 100 mW for excitation of the fiber, a broad emission band of Er around 1550 nm was observed as presented in Fig. 8.

In order to determine effects of ZrO_2 and BaO on the formation of nanocrystalline particles in silica materials, a preform without ZrO_2 and BaO doping was fabricated. Measured concentration profiles on this preform have revealed that the incorporated maximum Er concentration was by about 600 ppm lower than in the preform containing ZrO_2 and BaO. Moreover, it has been observed that the heat treatment of the preform without ZrO_2 and BaO induces neither any nanoparticles nor crystals within the preform core. This clearly indicates that the presented glass composition is a favorable one for the formation of the required nanocrystalline particles and able to incorporate elevated concentrations of Er.

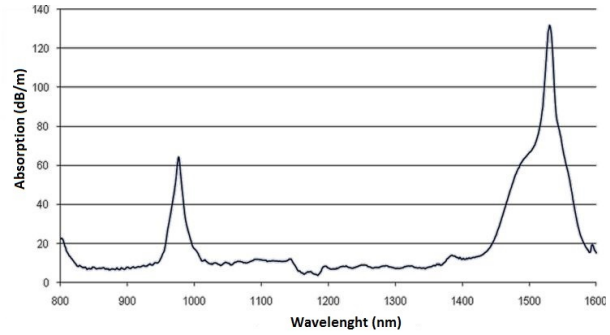

Fig. 7: Spectral attenuation losses of the prepared fiber.

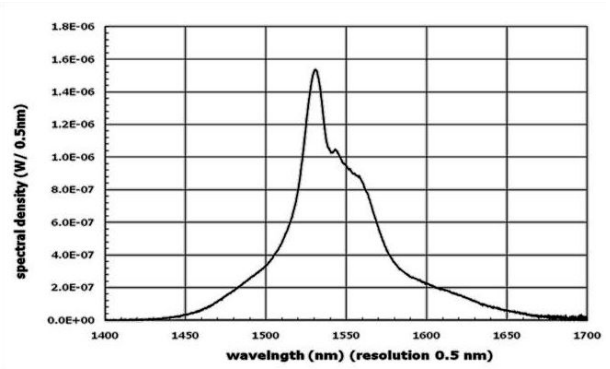

Fig. 8: Fluorescence spectra of the fabricated Er-doped fiber.

5. Conclusion

Preforms of silica-based Er-doped optical fibers have been fabricated using the MCVD method and solution doping techniques. The core of the preform contains two phase-separated regions. Er-doped ZrO_2-based nanocrystalline particles of average dimensions within $10 - 80$ nm are detectable by scanning electron microscopy in the preform core which is nearly transparent in spite of high Al_2O_3 ($\sim$14 mol%) doping levels and the presence of nanocrystalline particles. HRTEM result of fiber core sample clearly establish that nanocrystalline particles formed within preform core via post-processing, survive within fiber core and withstand the fiber drawing condition. The fabricated fiber exhibits a broad emission band of Er around 1550 nm evident from the measured fluorescence spectra. The incorporation of high Er concentration of about 0,48 mol% is possible for the selected glass composition. The variation of nanocrystalline particle sizes is expected to be controlled by a fine tuning of fabrication conditions on which we are currently working.

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